Docket No.: J-2605A

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: Richard W. Avery

Serial No.: - - - - - - -

Filed: Herewith

Title: PRODUCTION OF STABLE HYDROLYZABLE ORGANOSILANE SOLUTIONS

Commissioner for Patents
Washington, D.C. 20231

Letter under 37 C.F.R. 1.608(b)

Sir:

Enclosed herewith are a Declaration under 37 C.F.R. 1.608(b) of Richard W. Avery, applicant herein, with attached Exhibit A, and a corroborating declaration from a non-inventor witness, Ian C. Callaghan, in support of the attached Request for Interference with U.S. Patent No. 6,113,815 issued on September 5, 2000.

The Applicant's showing is adequate to establish at least a *prima facie* case of priority of invention under 37 C.F.R. §1.608(b). In this regard, it has been stated by the Court of Appeals for the Federal Circuit in *Hahn v. Wong*, 892 F.2d 1028, 1032 (CAFC 1989) that

> To establish reduction to practice of a chemical composition, it is sufficient to prove that the inventor actually prepared the composition and knew it would work.

The Applicant has established a *prima facie* case entitling him to proceed with the interference because the critical reference date of U.S. Patent No. 6,113,815 is no earlier than July 18, 1997 (the filing date of the provisional application from which U.S. Patent No. 6,113,815 claims benefit) and the Applicant's declaration shows evidence of his reduction to practice of the claims of the present application having interfering subject matter substantially

corresponding to claims 1-14, 16-24, 26-27, and 29-31 of U.S. Patent No. 6,113,815 in the United Kingdom (a WTO country since January 1, 1995) prior to July 18, 1997. In particular, the documents evidence that he actually prepared the compositions of the claims of the present application, tested that they would work for the desired utility, and appreciated the result, at least as early as January 23, 1997.

Specifically, looking at Exhibit A, page 2 that is attached to the Declaration under 37 C.F.R. 1.608(b) of Richard W. Avery, the inventor of the present application, there is shown a summary of experiments performed by the inventor and/or others working under his supervision. Along the left hand side of the summary of experiments, there are row headings for the Chemicals used in the compositions and for the Test Results, including a row heading entitled "Date made". Looking at the data summary under Reference 909EU58C', it can be seen that on or before December 3, 1996, the inventor and/or others working under his supervision had prepared a composition by mixing (among other things) 0.18% Fluorosilane 3M FC 405/60 and 5.00% propylene glycol n-butyl ether (which is widely known to have the formula $H\text{-}(CH_2)_4\text{-}O\text{-}(CH_2)_3\text{-}OH$). Likewise, under Reference 909EU58C'', it is shown that on or before December 3, 1996, the inventor and/or others working under his supervision had prepared a composition by mixing (among other things) 0.18% Fluorosilane 3M FC 405/60 and 2.50% propylene glycol n-butyl ether. Similarly, under Reference 909EU58C''', it is shown that on or before December 3, 1996, the inventor and/or others working under his supervision had prepared a composition by mixing (among other things) 0.18% Fluorosilane 3M FC 405/60 and 7.50% propylene glycol n-butyl ether.

Turning to Exhibit A, page 1 that is attached to the Declaration under 37 C.F.R. 1.608(b) of Richard W. Avery, the inventor specifically listed and attached to the original invention

disclosure the 3M Technical Bulletin that comprises pages 3-9 of Exhibit A. The 3M FC 405/60 organosilane is a fluor aliphatic silyl ether available from 3M Industrial Chemical Products. It is stated to have the general formula Rf-A-Si $(OMe)_3$, where Rf is a fluoroaliphatic group, and A is a linking group. More specifically, the active ingredient is 1-octanesulphonamide, N-ethyl-1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8-heptadecafluoro-N- 3-(trimethoxy silyl) propyl.

Looking at page 5 of Exhibit A, the hydrolysis of the three (OMe) groups on the Si atom of the 3M FC 405/60 organosilane with 3 H_2O molecules is clearly shown. The Rf-A- group on the Si atom is not shown as undergoing hydrolysis with H_2O molecules. Thus, the 3M FC 405/60 organosilane is properly described as an organosilane having three hydrolyzable groups (OMe) and one non-hydrolyzable organic group (Rf-A-).

Referring back to Exhibit A, page 2 that is attached to the Declaration under 37 C.F.R. 1.608(b) of Richard W. Avery, the summary of experiments shows that the inventor also recorded the initial stability and the stability after one month of the compositions, and the initial hydrophobic effect on glass and the hydrophobic effect on glass after the compositions aged for 1 month. Glass treatment is a desired utility for these components.

Turning to Exhibit A, page 1, the inventor reported that based on the test results "2.5%, 5% and 7.5% levels of the sparingly soluble PnB [propylene glycol n-butyl ether] in formulas 909EU58C', 909EU58C'', 909EU58C''' give good stabilisation and the fluoro-silane is still available for tethering after 1 month, [and it] should be noted that the hazy appearance of some of these formulas is not necessarily a negative for stability and the appearance could range from clear to hazy". Thus, the inventor had appreciated by that date that the compositions of formulas 909EU58C', 909EU58C'', 909EU58C''' were stable and suitable for glass conditioning even after being stored for one month.

Now, comparing claims 1, 2 and 3 of the present application to the mixtures 909EU58C', 909EU58C'', 909EU58C''' prepared in December 1996 by the inventor, it is clear that claims 1 and 2 of the present application both read on the mixtures 909EU58C', 909EU58C'', 909EU58C''' prepared in December 1996 by the inventor and that the mixtures 909EU58C', 909EU58C'', 909EU58C''' read on at least one species of claim 3. See Tables 1, 2 and 3 below.

Table 1

Term in Claim 1 of the Present Application	Support in Invention Disclosure of Inventor's Declaration
a) an organosilane of the formula R_nSiX_{4-n}, wherein n is an integer of from 0 to 3; and R is, independently, a nonhydrolyzable organic group, and each X is, independently, a hydrolyzable group	When n=1, the organosilane in the claim is R_1SiX_3 where R is a nonhydrolyzable organic group, and X is a hydrolyzable group. The 3M FC 405/60 organosilane used in mixtures 909EU58C', 909EU58C'', 909EU58C''' of the invention disclosure has the general formula R_fA-Si-$(OMe)_3$ where R_fA is a nonhydrolyzable organic group, and OMe is a hydrolyzable group. This is clearly supported by the 3M Technical Bulletin that was an integral part of the invention disclosure. Thus, the 3M FC 405/60 organosilane meets the general formula R_1SiX_3 where R is a nonhydrolyzable organic group, and X is a hydrolyzable group.
b) an ether of the formula R-O-R, wherein R is, independently, an organic group, and the ether has a hydroxy functionality.	The composition of the invention disclosure uses propylene glycol n-butyl ether which has the formula $H-(CH_2)_4-O-(CH_2)_3-OH$. The R groups of propylene glycol n-butyl ether (i.e., $H-(CH_2)_4$-- and $-(CH_2)_3-OH$) are organic groups and there is one hydroxyl (-OH) group in the ether.

Table 2

Term in Claim 2 of the Present Application	Support in Invention Disclosure of Inventor's Declaration
a) an organosilane of the formula R_nSiX_{4-n}, wherein n is an integer of from 0 to 3; and R is, independently, a nonhydrolyzable organic group, and each X is, independently, a hydrolyzable group	When n=1, the organosilane in the claim is R_1SiX_3 where R is a nonhydrolyzable organic group, and X is a hydrolyzable group. The 3M FC 405/60 organosilane used in mixtures 909EU58C', 909EU58C'', 909EU58C''' of the invention disclosure has the general formula R_fA-Si-$(OMe)_3$ where R_fA is a nonhydrolyzable organic group, and OMe is a hydrolyzable group. This is clearly supported by the 3M Technical Bulletin that was an integral part of the invention disclosure. Thus, the 3M FC 405/60 organosilane meets the general formula R_1SiX_3 where R is a nonhydrolyzable organic group, and X is a hydrolyzable group.
b) an ether of the formula R-O-R, wherein R is, independently, an organic group, and the ether has a hydroxy functionality,	The composition of the invention disclosure uses propylene glycol n-butyl ether which has the formula H-$(CH_2)_4$-O-$(CH_2)_3$-OH. The R groups of propylene glycol n-butyl ether (i.e., H-$(CH_2)_4$-- and -$(CH_2)_3$-OH) are organic groups and there is one hydroxyl (-OH) group in the ether.
wherein the ether is a glycol ether	The composition of the invention disclosure uses propylene glycol n-butyl ether which is a glycol ether.

Table 3

Term in Claim 3 of the Present Application	Support in Invention Disclosure of Inventor's Declaration
wherein the ether is selected from dipropylene glycol butyl ether, propylene glycol butyl ether, and dipropylene glycol propyl ether.	The composition of the invention disclosure uses propylene glycol n-butyl ether.

It is respectfully submitted that the foregoing analysis of the Declaration under 37 C.F.R. 1.608(b) of Richard W. Avery, applicant herein, shows that the inventor actually prepared the composition of claims 1 and 2 of the present application and at least a species of claim 3, tested that the compositions would work for a desired utility, and appreciated the successful results, at

least as early as January 23, 1997, all in the United Kingdom. Because the inventor of the above-referenced application reduced to practice the invention of claims 1 and 2 and a species of claim 3 of the present application prior to the critical reference date of U.S. Patent No. 6,113,815 (July 18, 1997), the Applicant is *prima facie* entitled to an award of priority over the interfering claims of U.S. Patent No. 6,113,815.

Respectfully submitted,

Dated: 8/31, 2001

By: _____

J. William Frank, III
Registration No. 25,626
S.C. Johnson & Son, Inc.
Legal Department
1525 Howe Street
Racine, WI 53403
(262) 260-2673

QBMKE 5106381.3

Docket No.: J-2605A

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: Richard W. Avery

Serial No.: - - - - - - -

Filed: Herewith

Title: PRODUCTION OF STABLE HYDROLYZABLE ORGANOSILANE SOLUTIONS

Commissioner for Patents
Washington, D.C. 20231

Corroborating Witness Declaration Under 37 C.F.R. 1.608(b)

Sir:

I, Ian C. Callaghan, declare that:

1. I was employed as Global Platform Leader, Surfactants, Polymers and Emulsions at S.C. Johnson Wax, Milton Park, United Kingdom from January 1, 1994 to June 30, 1998. During January of 1997 (and at other times), I worked with Richard W. Avery.

2. At least as early as January 1997, I became aware in the United Kingdom of Richard W. Avery's work regarding the glycol ether stabilization of organosilanes including his reduction to practice in the United Kingdom of the attached claimed subject matter.

3. My knowledge of Richard W. Avery's work regarding the ether stabilization of organosilanes is confirmed by my signature near the bottom left of the attached Exhibit A invention disclosure. Exhibit A is an accurate photocopy of the Invention Disclosure that I witnessed Richard W. Avery sign on January 25, 1997 in the United Kingdom (albeit the

"EL97780" tracking number, the NI "2605" number, and the submission approval signature were later added).

4. I am making this statement in corroboration of his attached Declaration under 37 C.F.R. 1.608(b).

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Respectfully submitted,

Dated: August 27th, 2001

By: Ian C. Callaghan
Ian C. Callaghan

Claims Pending In The Above-Identified Patent Application

1. A composition comprising a mixture of:

a) an organosilane of the formula R_nSiX_{4-n}, wherein n is an integer of from 0 to 3; and R is, independently, a nonhydrolyzable organic group, and each X is, independently, a hydrolyzable group; with

b) an ether of the formula R-O-R, wherein R is, independently, an organic group, and the ether has a hydroxy functionality.

2. A composition comprising a mixture of:

a) an organosilane of the formula R_nSiX_{4-n}, wherein n is an integer of from 0 to 3; and R is, independently, a nonhydrolyzable organic group, and each X is, independently, a hydrolyzable group; with

b) an ether of the formula R-O-R, wherein R is, independently, an organic group, and the ether has a hydroxy functionality,

wherein the ether is a glycol ether.

3. The composition of claim 2, wherein the ether is selected from dipropylene glycol butyl ether, propylene glycol butyl ether, and dipropylene glycol propyl ether.

Docket No.: J-2605A

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Applicant: Richard W. Avery

Serial No.: - - - - - - -

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Title: PRODUCTION OF STABLE HYDROLYZABLE ORGANOSILANE SOLUTIONS

Commissioner for Patents
Washington, D.C. 20231

Request for Interference under 37 C.F.R. § 1.607 Accompanying Application

Sir:

It is requested that an interference be declared with enclosed U.S. Patent No. 6,113,815 issued on September 5, 2000 based on the following claims 1-3 that were filed with the above-referenced application.

1. A composition comprising a mixture of:

a) an organosilane of the formula R_nSiX_{4-n}, wherein n is an integer of from 0 to 3; and R is, independently, a nonhydrolyzable organic group, and each X is, independently, a hydrolyzable group; with

b) an ether of the formula R-O-R, wherein R is, independently, an organic group, and the ether has a hydroxy functionality.

2. A composition comprising a mixture of:

a) an organosilane of the formula R_nSiX_{4-n}, wherein n is an integer of from 0 to 3; and R is, independently, a nonhydrolyzable organic group, and each X is, independently, a hydrolyzable group; with

b) an ether of the formula R-O-R, wherein R is, independently, an organic group, and the ether has a hydroxy functionality,

wherein the ether is a glycol ether.

3. The composition of claim 2, wherein the ether is selected from dipropylene glycol butyl ether, propylene glycol butyl ether, and dipropylene glycol propyl ether.

Source Of The Claims Filed In The Above-Referenced Application

(1) Claim 1 of the above-referenced application has been substantially copied from claim 1 of U.S. Patent No. 6,113,815 with the deletion of the alternative ether carboxylic ester functionality. The exact changes to claim 1 of U.S. Patent No. 6,113,815 that resulted in Claim 1 of the above-referenced application are indicated below with brackets [ ] showing deletions.

- - 1. A composition comprising a mixture of:

a) an organosilane of the formula R_nSiX_{4-n}, wherein n is an integer of from 0 to 3; and R is, independently, a nonhydrolyzable organic group, and each X is, independently, a hydrolyzable group; with

b) an ether of the formula R-O-R, wherein R is, independently, an organic group, and the ether has [either] a hydroxy functionality [or carboxylic ester functionality]. --

(2) Claim 2 of the above-referenced application is an independent claim that limits the ether of the composition of Claim 1 to a glycol ether.

(3) Claim 3 of the above-referenced application further limits the glycol ether of the composition of Claim 2.

Suggestion for Counts

The Applicant suggests that a first Count could have the exact language of Claim 1 as filed with the above-referenced application and that a second Count could have the exact language of Claim 2 as filed with the above-referenced application.

Correspondence of Claims 1-14, 16-24, 26-27, and 29-31 of U.S. Patent No. 6,113,815 to Claims 1 and 2 of the Present Application

Claims 1-14, 16-24, 26-27, and 29-31 of U.S. Patent No. 6,113,815 correspond to claims 1 and 2 as follows. If a first Count were formulated having the exact language of claim 1 and a second count were formulated having the exact language of claim 2, the following also would describe correspondence to the counts.

Claim 1 of U.S. Patent No. 6,113,815 substantially corresponds to Claim 1 of the present application as shown above in the "Basis Of The Claims Filed In The Above-Referenced Application" section, i.e., with the deletion of the alternative ether carboxylic ester functionality.

Claim 2 of U.S. Patent No. 6,113,815 corresponds to Claim 1 of the present application in that it merely limits the organosilanes of the composition.

Claim 3 of U.S. Patent No. 6,113,815 corresponds to Claim 1 of the present application when the ether is other than a glycol ether in that it merely limits the ethers of the composition.

Claim 3 of U.S. Patent No. 6,113,815 corresponds to Claim 2 of the present application when the ether is a glycol ether.

Claim 4 of U.S. Patent No. 6,113,815 corresponds to Claim 1 of the present application in that it merely limits the ethers of the composition.

Claim 5 of U.S. Patent No. 6,113,815 corresponds to Claim 1 of the present application in that it merely limits the organosilanes of the composition.

Claim 6 of U.S. Patent No. 6,113,815 corresponds to Claim 1 of the present application in that it merely further limits the organosilanes of the composition.

Claim 7 of U.S. Patent No. 6,113,815 corresponds to Claim 1 of the present application in that it merely further limits the organosilanes of the composition.

Claim 8 of U.S. Patent No. 6,113,815 corresponds to Claim 1 of the present application in that it adds water to the composition and Claim 1 of the present application uses a "comprising" transition phrase.

Claim 9 of U.S. Patent No. 6,113,815 corresponds to Claim 1 of the present application in that it merely limits the organosilanes of the composition.

Claim 10 of U.S. Patent No. 6,113,815 corresponds to Claim 1 of the present application in that it merely limits the use of the composition.

Claim 11 of U.S. Patent No. 6,113,815 corresponds to Claim 1 of the present application in that it merely adds a carrier other than water to the use of the composition and Claim 1 of the present application uses a "comprising" transition phrase.

Claim 12 of U.S. Patent No. 6,113,815 corresponds to Claim 1 of the present application in that it merely limits the organosilanes of the composition.

Claim 13 of U.S. Patent No. 6,113,815 corresponds to Claim 1 of the present application in that it merely limits the organosilanes of the composition.

Claim 14 of U.S. Patent No. 6,113,815 corresponds to Claim 1 of the present application in that it merely limits the organosilanes of the composition.

Claim 16 of U.S. Patent No. 6,113,815 corresponds to Claim 1 of the present application in that it merely limits the organosilanes of the composition.

Claim 17 of U.S. Patent No. 6,113,815 corresponds to Claim 1 of the present application in that it merely limits the organosilanes of the composition.

Claim 18 of U.S. Patent No. 6,113,815 corresponds to Claim 1 of the present application in that it merely limits the organosilanes of the composition.

Claim 19 of U.S. Patent No. 6,113,815 corresponds to Claim 1 of the present application in that it merely adds water to the composition and Claim 1 of the present application uses a "comprising" transition phrase.

Claim 20 of U.S. Patent No. 6,113,815 corresponds to Claim 1 of the present application in that it recites an inherent use of the composition.

Claim 21 of U.S. Patent No. 6,113,815 corresponds to Claim 1 of the present application in that it recites an inherent use of the composition.

Claim 22 of U.S. Patent No. 6,113,815 corresponds to Claim 1 of the present application in that it recites an inherent use of the composition.

Claim 23 of U.S. Patent No. 6,113,815 corresponds to Claim 1 of the present application in that it recites an inherent use of the composition.

Claim 24 of U.S. Patent No. 6,113,815 corresponds to Claim 1 of the present application in that it recites an inherent use of the composition.

Claim 26 of U.S. Patent No. 6,113,815 corresponds to Claim 1 of the present application in that it recites an inherent use of the composition.

Claim 27 of U.S. Patent No. 6,113,815 corresponds to Claim 1 of the present application in that it recites an inherent use of the composition.

Claim 29 of U.S. Patent No. 6,113,815 corresponds to Claim 1 of the present application in that it limits the amount of organosilane and ether and merely adds water to the composition and Claim 1 of the present application uses a "comprising" transition phrase.

Claim 30 of U.S. Patent No. 6,113,815 corresponds to Claim 1 of the present application in that it merely limits the ethers of the composition.

Claim 30 of U.S. Patent No. 6,113,815 corresponds to Claim 2 of the present application when the ether of claim 30 is a glycol ether.

Claim 31 of U.S. Patent No. 6,113,815 corresponds to Claim 1 of the present application in that it recites an inherent use of the composition.

Support for the Terms in Claims 1, 2 and 3 of the Present Application

The terms of the claims of the application are supported in the Applicant's specification as shown in the following Table 1.

Table 1

Term In Claims of the Present Application			Supporting Language in Present Specification	
Claim No.	Line Nos.	Term	Page No.	Line Nos.
1	1	Mixture	2	4
1	2-4	an organosilane of the formula R_nSiX_{4-n}, wherein n is an integer of from 0 to 3; and R is, independently, a nonhydrolyzable organic group, and each X is, independently, a hydrolyzable group	3 and 3 and 7	2-5 20-28 21-22
1	5-6	an ether of the formula R-O-R, wherein R is, independently, an organic group, and the ether has a hydroxy functionality	7	17-18

Term In Claims of the Present Application			Supporting Language in Present Specification	
2	1	Mixture	2	4
2	2-4	an organosilane of the formula R_nSiX_{4-n}, wherein n is an integer of from 0 to 3; and R is, independently, a nonhydrolyzable organic group, and each X is, independently, a hydrolyzable group	3 and 3 and 7	2-5 20-28 21-22
2	5-6	an ether of the formula R-O-R, wherein R is, independently, an organic group, and the ether has a hydroxy functionality	7	17-18
2	7	Wherein the ether is a glycol ether	7 and Abstract	17-18 line 3
3	1-2	wherein the ether is dipropylene glycol butyl ether, propylene glycol butyl ether, or dipropylene glycol propyl ether	4	21-22

Compliance with Certain Time Limits

A claim has been made in the present application which is substantially the same subject matter as a claim of U.S. Patent No. 6,113,815 prior to one year from the date on which U.S. Patent No. 6,113,815 was granted (September 5, 2000).

Papers complying with 37 C.F.R. Section 1.608(b) have been provided with this request.

Respectfully submitted,

Dated: 8/31, 2001

By: _____

J. William Frank, III
Registration No. 25,626
S.C. Johnson & Son, Inc.
Legal Department
1525 Howe Street
Racine, WI 53403
(262) 260-2673

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Title: PRODUCTION OF STABLE HYDROLYZABLE ORGANOSILANE SOLUTIONS

Commissioner for Patents
Washington, D.C. 20231

Inventor's Declaration Under 37 C.F.R. 1.608(b)

Sir:

I, Richard W. Avery, the named inventor of the above-identified patent application, hereby declare as follows:

1. I conceived and reduced to practice in the United Kingdom prior to July 18, 1997 the invention specified in the attached claims which I understand are those pending in the above-identified patent application and which I understand are believed to correspond to the proposed counts.

2. This is evidenced by attached Exhibit A, an accurate photocopy of an Invention Disclosure that I signed on 23-January-1997 in the United Kingdom (albeit the "EL97/80" tracking number, the NI "2605" number, and the submission approval signature were later added). Page 2 of Exhibit A shows formulas that I, or others acting under my supervision, prepared in November and December 1996 in the United Kingdom. It is an accurate summary

created at least as early as January 23,1997 in the United Kingdom of tests I, or others acting under my supervision, performed.

3. The Exhibit A Invention Disclosure confirms that I prepared in December 1996 a composition by mixing (among other things) an organosilane with three hydrolyzable groups and one nonhydrolyzable group (3M FC 405/60) and propylene glycol n-butyl ether, which has the formula $H-(CH_2)_4-O-(CH_2)_3-OH$. The 3M FC 405/60 organosilane (as shown in the 3M Technical Bulletin specifically referenced by me in the Invention Disclosure) is a fluor aliphatic silyl ether available from 3M Industrial Chemical Products. It is stated to have the general formula Rf-A-Si $(OMe)_3$, where Rf is a fluoroaliphatic group, and A is a linking group. More specifically, the active ingredient is 1-octanesulphonamide, N-ethyl-1,1,2,2,3,3,4,4,5,5,6,6,7,7,8,8-heptadecafluoro-N- 3-(trimethoxy silyl) propyL I recorded the physical and chemical stability of the mixtures, and their utility for glass conditioning.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Respectfully submitted,

Dated: August 23, 2001

By: R. W. Avery
Richard W. Avery

EL97/80

INVENTION DISCLOSURE

(LEAVE BLANK)

Date Received _____

NI 26C5

J1036 U.S. PTO 09/945142 08/31/01

Instructions: ***Use this form as an initial disclosure the Law Department. Preliminary patentability searches will be made based on the contents of this disclosure, so answer the questions fully.***

EXHIBIT A Page 1

Date 23 January 1997

1 Inventor(s) R. W. Avery

2 Type of Disclosure Formulation

3 Brief description of invention Improvements to the stabilisation of silanes, including fluoro-silanes in aqueous formulations.

4 In the space below and/or on an attached sheet, describe the essential elements or steps of your invention, using chemical names to describe formulation ingredients; the function of each essential element, component, or step; the optimum value and the upper and lower limits for each pertinent parameter, such as temperature, time, pressure, pH, and concentration(s), amounts or ratios, amount of essential components, the effect of varying each parameter, and optional ingredients, elements or process steps, including amount and function. Also, list any possible substitution(s) for each essential element and use a drawing or flow sheet, if appropriate.

Improvements to the Stabilisation of Silanes, including fluoro-silanes, in Aqueous solutions

Relevant Prior SC Art -

U.S. patent 5,411,585 (Avery et al.) describes ways to stabilise a water soluble organo silane

Invention disclosure NI 2406 (Clare Norman) describes stablisation of organosilanes and fluorosilanes.

Invention -

U.S patent is useful in stabilising organo silanes but if less soluble or insoluble silanes and fluorosilanes are being used it is difficult to get adequate stability. Cosolvents have been suggested previously as useful for stabilising silanes in aqueous solutions, but only at excessive levels.

Suprisingly it has been found that low levels of sparingly water soluble solvents such as propylene glycol n-butyl ether or dipropylene glycol n-butyl ether are very efficient at low levels 12% or less in stabilising less soluble or insoluble silanes (or fluorosilanes) in modified systems taken from U.S. 5,411,585 (quaternary, nonionic or sarcosinate co-surfactants) and NI 2406 (alkyl polyglucosides).

From the attached formula summary document the following conclusions were drawn -

a) The formula with no solvent, 909EU42E, was very unstable and immediately separated.
b) The formula with water soluble solvent Dowanol PM, 909EU56C, was initially stable and gave strong hydrophobic conditioning to glass, but unfortunately it is not stable, as the fluoro-silane is not available for hydrophobic conditioning after 1 month.
c) 2.5%, 5% and 7.5% levels of the sparingly soluble PnB in formulas 909EU58C', 909EU53C'' and 909EU53C''' give good stabilisation and the fluoro-silane is still available for tethering after 1 month. It should be noted that the hazy appearance of some of these formulas is not necessarily a negative for stability and the appearance could range from clear to hazy
d) A suprising aspect of this invention is that the higher level of PnB is much less effective at stabilising the fluoro-silane than the lower levels discussed in c)

The use of sparingly solvents also broadens out the range of surfactants which can be used. In summary the less soluble or insoluble silanes and fluorosilanes can be stabilised by combining with them in the aqueous formulation with sparingly soluble solvents, plus surfactants such as quaternary surfactants, nonionic surfactants (including alkyl glucosides) and amphoterics.

List and attach copies of any patents, publications, technical reports, or supplier brochures which relate to the above invention.

US patent 5,411,585 (Avery et al.) and Invention disclosure NI 2406 (Clare Norman), which were already discussed - see above.

SM Technical data document issued July 15, 1994 - This mentions in section III, Application sub section 2. that aqueous solution pot-life is only 8-16 hours

Each person listed in No.1 above must sign and date this form in the space below and have that signature witnessed by their manager.

WITNESS(ES) INVENTOR(S)

J. C. Ballagh — R. W. Avery — DATE 23-January 1997

_____ _____ DATE _____

_____ _____ DATE _____

Approved for submission _____ 26/1/97 Director, R.D&E Europe

Acting to be via P.D. Allen then I.T. Ryan

Improvements to the Stabilisation of Silanes in Aqueous Solution.

(R. W. Avery / J. C. Beesley - 23 January 1997)

Code	Chemical	Reference	909EU42E	909EU56C	909EU56B	909EU58C'	909EU58C''	909EU58C'''
			%	%	%	%	%	%
336701	Deionised Water		95.46	85.46	85.46	90.46	92.96	87.96
342211	Empigen BAC (50%)		2.00	2.00	2.00	2.00	2.00	2.00
342777	Lutensol AO8		2.00	2.00	2.00	2.00	2.00	2.00
	Zonyl FSD (30%)		0.36	0.36	0.36	0.36	0.36	0.36
348516	Lactic Acid		<---------- Adjust pH to approx. 4 with Lactic Acid ---------->					
346004	Propylene Glycol n-Butyl Ether (PnB)			10.00		5.00	2.50	7.50
336011	Propylene Glycol Methyl Ether (Dowanol PM)				10.00			
	Fluoro Silane 3M FC 405/60		0.18	0.18	0.18	0.18	0.18	0.18
			100.00	100.00	100.00	100.00	100.00	100.00

Test Results

Date made	25-Nov-96	02-Dec-96	02-Dec-96	03-Dec-96	03-Dec-96	03-Dec-96
Initial stability	formula separates	OK	slightly hazy	slightly hazy	hazy	hazy
Stability after 1 month at ambient temperature	not applicable	separates	separates	slightly hazy	hazy	hazy
Stability after 1 month at 40 deg C	not applicable	separates	separates	OK	precipitates	particles in solution
Initial Hydrophobic affect (test on glass and after detergent rinse)	not applicable	strong	moderate	strong	strong	very strong
Hydrophobic affect after formulas aged 1 month (test on glass and after detergent rinse)	not applicable	nil	nil	fairly strong	fairly strong	strong

technical data

TITLE FC-405-60

FLUORAD™

DATE ISSUED JULY 15, 1994
SUPERSEDES ISSUE OF

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Product Information

The chemical FC-405-60 can be applied to glass, ceramics and other silicones or silicate surfaces to provide low surface energy films. The treated glass will have enhanced scratch resistance, good lubricity, oil- and waterrepellency, and release properties. Alternatively FC-405-60 can be used to promote adhesion between these surfaces and PTFE-based polymers.

Some examples are :

1. Surface Protection

FC-405-60 can be used alone or in formulation with other chemicals to treat the surfaces of containers, light bulbs, reaction vessels, and other glass articles to give scratch resistance, toughness and lubricity.

2. Release coatings

Many resins will not adhere to glass which has been treated with FC-405-60. Epoxies, acrylics and many other resins, as well as adhesives, will release easily from the treated glass or ceramic substrate.

Pressure sensitive tapes, especially after aging in sunlight, will release more easily from FC-405-60 treated glass.

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3. Oil- and waterrepellency

This chemical can be used to obtain repellency of any liquid having a surface tension greater than about 15 dynes/cm. This includes water, oils, ketones, gasoline and aromatic and aliphatic hydrocarbons.

4. Adhesion promotion

In the production of glassfiber reinforced PTFE, FC-405-60 can serve as an adhesion promotor. Being chemically bound to the glass fiber on the one side and through its perfluorinated tail being compatible with the PTFE polymer on the other side, improved adhesion of the PTFE polymer onto the glass fiber is achieved.

I. Typical physical properties (not for specification purposes)

FC-405-60 can be described as a solution of fluoroaliphatic silyl ethers.

Appearance	Yellow liquid
Specific gravity (25°C)	+/- 1.2
Flash point	12°C
Type	Nonionic
Composition	60% fluorochemical
	40% ethanol

FC-405-60 can be diluted with alcohols, ketones, acetates, ethers, ...
FC-405-60 can also be dispersed in water.

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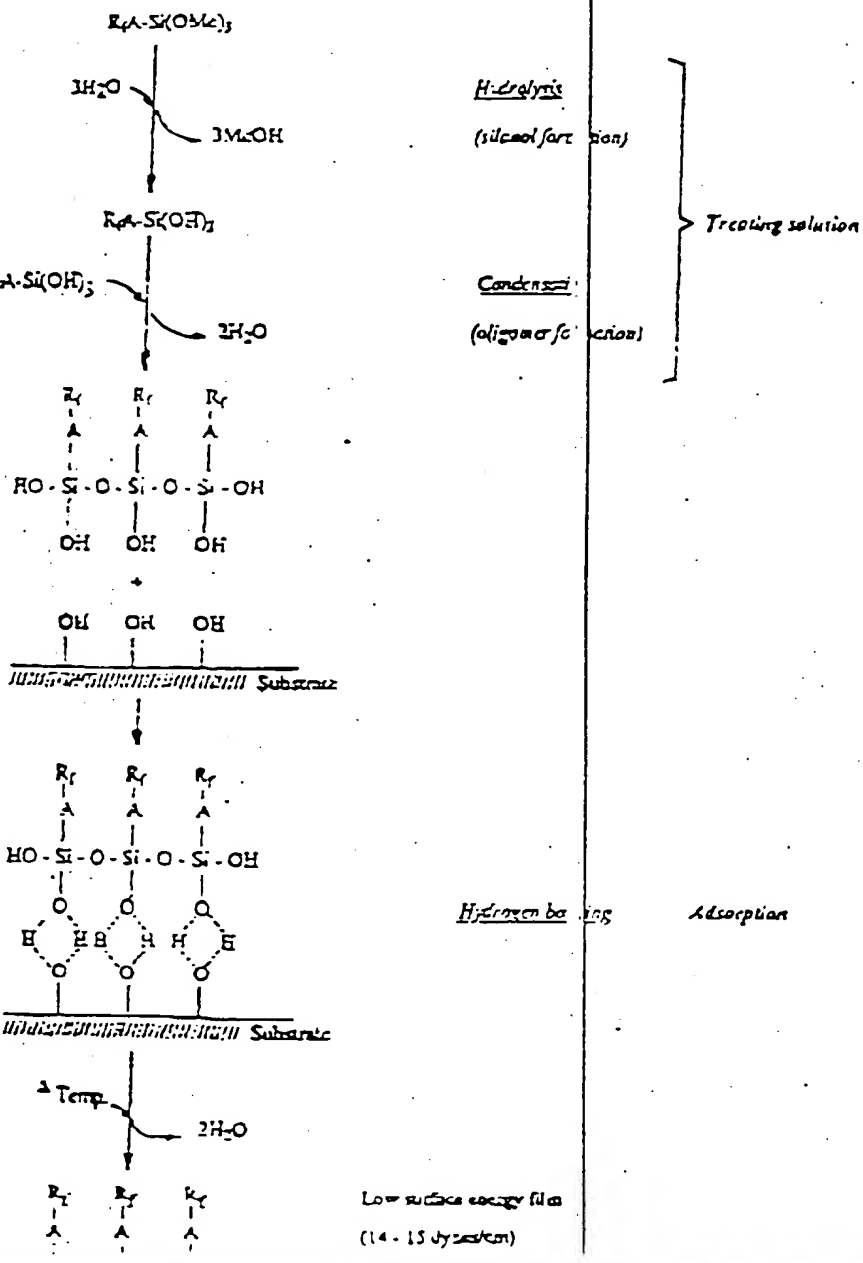
II. Description

FC-405-60 is a concentrated solution of fluoroaliphatic silyl ethers. It will react with siliceous surfaces to create a low surface energy film that cannot be wetted with liquids. The film is stable and will not be removed except by conditions or reagents that attack the glass. Properly applied films will have a critical surface energy of 14-15 dynes/cm. The reaction mechanism can generally be described as follows :



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III. Application

FC-405-60 is a concentrated stock-solution in eth nol and should be further diluted to an experimentally defined working solution suitable or the particular application. This is done by adding FC-405-60 to the desired amount f a suitable solvent for that application, selected from alcohols, ketones, acet es, ethers.
The optimal treatment level may vary from 0.02- 00% by weight of active material.

It is important that the surface to be treated is clea and free of other treatments. FC-405-60 cannot be applied over oily or silicone or stear e treated glass. Ideally, the treatment can be applied to glass or ceramics freshly emerg from the annealing lehr as is done in many instances. If not possible, cleaning of the s bstrate can be done using hot solutions of a suitable wetting agent followed by sufficient nsing with distilled water. Additional drying is preferred but not always necessary.
The following methods for preparing fluorosilyla d surfaces are based on a standard treatment level of 0.5% active material and can b considered a good starting p point for determination of the optimal concentration.

1. Deposition from aqueous alcohol solutions

An ethanol/water solution (95/5) is adjusted to H 4.5-5.5 with acetic acid.
1 part of FC-405-60 is added to 119 parts (by w ight) of this solution with stirring to yield a 0.5% final concentration.
Allow 5 minutes for hydrolysis and silanol for ation.

Adsorption of the chemical onto the substrate (ydrogen bonding) :

a) Large objects, e.g. glass plates, are dippe nto the solution, agitated gently and removed after 1-2 minutes.
They are rinsed free of excess material by pping briefly in ethanol.

b) Particles, e.g. fillers and supports, are trea d by stirring them in the solution for 2-3 minutes and then decanting the solution. The particles are then rinsed twice briefly with ethanol.

Curing of the fluoroaliphatic layer is done at 11 °C for 5-10 min. or for 24 hrs at room temperature (< 60% R.H.).

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2. Deposition from aqueous solution/dispersio

This method is employed for most commer al fiberglass systems.

Dissolve 1 part of FC-405-60 in 119 parts c a mixture (25/95 by weight) of ethanol/water to yield a 0.5% final concentr tion. Adequate stirring should be used to insure proper mixing. The resulting hazy dispersion has a useful pot-life of 10-16 hrs.

If solubility is not sufficient, 0.1% of a non- onic surfactant is added prior to addition of FC-405-60 and an emulsion rath r than a solution is prepared.

The solution is adjusted to pH 5.5 with acet acid.

This treatment solution can either be spraye or brushed onto the substrate or employed as dip bath.

Allow 1-2 minutes contact of the solution w h the substrate and then rinse it with fresh distilled water and allow it to drain for couple of minutes.

Cure is done at 110-120°C for 5-10 minutes

3. Bulk deposition onto powders

E.g. filler treatment, can be accomplished b a spray-on method, provided the amount fluorochemical necessary is known d that sufficient adsorbed moisture is present on the filler to cause hydrolysis of th chemical.

The necessary amount is prepared as a 20% olution in ethanol by adding 1 part FC-405-60 to 2 parts of ethanol.

The powder is placed in a high intensity soli mixer, e.g. twin cone mixer with intensifier.

The solution is pumped into the agitated pow er as a fine spray.

Dynamic drying methods are most effective. If the filler is dried in trays, care must be taken to avoid wicking or skimming of the op layer of treated material by adjusting heat and air flow.

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IV. Durability

The fluoroaliphatic protective layer is not removed by :

1. Temperatures to 260°C in an oxidizing atmosphere; higher temperatures can be tolerated in non-oxidative environment.
2. Boiling solvents such as DMF, alcohol, acetone or water.
3. Boiling aqueous acids such as sulfuric, nitric, acetic.

Some reagents which will remove the surface properties are : 10% aqueous HF, boiling concentrated H_3PO_4, dilute NaOH at room temperature, and hot aqueous ammonia (partial removal).

V. Bibliography

1. Francen, V.L. and Heine, R.F., The Glass Industry, 46, p.594, (1965)

2. Noll W., Chemistry and Technology of Silicones. Academic Press Inc. (London) Ltd.

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